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(54) Electrophotographic elements containing new electron-transport agents.

(57) New electrophotographic elements contain electron-transport agents comprising certain new chemical compounds, which are derivatives of 4H-thiopyran-1,1-dioxides.

ELECTROPHOTOGRAPHIC ELEMENTS CONTAINING NEW ELECTRON-TRANSPORT AGENTS

This invention relates to electrophotographic elements containing electron-transport agents comprising certain chemical compounds, which are derivatives of 4H-thiopyran-1,1-dioxides. The chemical compounds have unexpectedly good solubility or dispersibility in organic solvents and polymeric binders, and they exhibit good electron-transport properties in the inventive electrophotographic elements.

In electrophotography an image comprising a pattern of electrostatic potential (also referred to as an electrostatic latent image), is formed on a surface of an electrophotographic element comprising at least an insulative photoconductive layer and an electrically conductive substrate. The electrostatic latent image is usually formed by imagewise radiation-induced discharge of a uniform potential previously formed on the surface. Typically, the electrostatic latent image is then developed into a toner image by contacting the latent image with an electrographic developer. If desired, the latent image can be transferred to another surface before development.

In latent image formation the imagewise discharge is brought about by the radiation-induced creation of electron/hole pairs, which are generated by a material (often referred to as a charge-generation material) in the electrophotographic element in response to exposure to the imagewise actinic radiation. Depending upon the polarity of the initially uniform electrostatic potential and the type of materials included in the electrophotographic element, either the holes or the electrons that have been generated migrate toward the charged surface of the element in the exposed areas and thereby cause the imagewise discharge of the initial potential. What remains is a non-uniform potential constituting the electrostatic latent image.

Many electrophotographic elements currently in use are designed to be initially charged with a negative polarity. Such elements contain material which facilitates the migration of positive holes toward the negatively charged surface in imagewise exposed areas in order to cause imagewise discharge. Such material is often referred to as a hole-transport agent. In elements of that type a positively charged toner material is then used to develop the remaining imagewise unexposed portions of the negative polarity potential (i.e., the latent image) into a toner image. Because of the wide use of negatively charging elements, considerable numbers and types of positively charging toners have been fashioned and are available for use in electrographic developers. Conversely, relatively few high quality negatively charging toners are available.

However, for some applications of electrophotography it is more desirable to be able to develop the surface areas of the element that have been imagewise exposed to actinic radiation, rather than those that remain imagewise unexposed. For example, in laser printing of alphanumeric characters it is more desirable to be able to expose the relatively small percentage of surface area that will actually be developed to form visible alphanumeric toner images, rather than waste energy exposing the relatively large percentage of surface area that will constitute undeveloped background portions of the final image. In order to accomplish this while still employing widely available high quality positively charging toners, it is necessary to use an electrophotographic element that is designed to be positively charged. Thus, positive toner can then be used to develop the exposed surface areas (which will have relatively negative electrostatic potential after exposure and discharge, compared to the unexposed areas, where the initial positive potential will remain).

An electrophotographic element designed to be initially positively charged should, however, contain an adequate electron-transport agent (i.e., a material which adequately facilitates the migration of photogenerated electrons toward the positively charged insulative element surface). Unfortunately (and analogous to the situation with positive and negative toners), many materials having good hole-transport properties have been fashioned for use in electrophotographic elements, but relatively few materials are known to provide good electron-transport properties in electrophotographic elements.

A number of chemical compounds having electron-transport properties are described, for example, in U.S. Patents 4,175,960; 4,514,481; 4,474,865; 4,559,287; 4,606,861; and 4,609,602. However, many prior art compounds have one or more drawbacks.

Some prior art electron-transport agents do not perform the electron-transporting function very well, especially under certain conditions or when included in certain types of electrophotographic elements. Also, some cause an undesirably high rate of discharge of the electrophotographic element before it is exposed to actinic radiation (often referred to as high dark decay).

Furthermore, some prior art electron-transport compounds are not soluble or dispersible or have relatively limited solubility or dispersibility in coating solvents of choice or in some polymeric binders desired to be used in charge-transport layers, such that attempts to include sufficient amounts of such electron-transport agents in electrophotographic elements result in some crystallization of the electron-transport agents, which in turn causes problems such as undesirable levels of dark decay and such as unwanted scatter or absorption of actinic radiation intended to pass undisturbed through the charge-transport layer to a radiation-sensitive portion of the element.

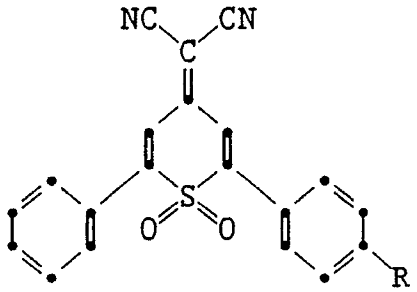
Even when sufficient amounts of electron-transport agent for adequate performance can be initially compatibly incorporated in an electrophotographic element, problems can arise thereafter during use of the element. For example, U.S. Patent 4,514,481 describes a number of specific electron-transport agents (e.g., 4-dicyanomethylene-2,6-diphenyl-4H-thiopyran-1,1-dioxide) and illustrates incorporating them in polymeric binder layers of electrophotographic elements at a concentration of 30% by weight (based on total weight of the agent and the binder) for good performance. However, in fact, the upper limit of compatibility (solubility or homogeneous dispersibility) of compounds such as 4-dicyanomethylene-2,6-diphenyl-4H-thiopyran-1,1-dioxide in many polymeric binders is about 35% by weight. At such concentration the compounds are on the edge of incompatibility. At elevated temperatures, such as the element can encounter during normal use in a copier, the compound can more easily migrate within the binder and tend to form crystalline agglomerates that cause problems such as noted above.

Even if such a problem does not occur, if it is desired to increase the concentration of such an electron-transport agent beyond 35% by weight, there is no leeway to do so. For example, it is well known in the art that increasing the concentration of an electron-transport agent in a polymeric layer, without phase-separation, increases the electron-transport mobility of the layer; i.e., photogenerated electrons will move through the layer at a higher velocity and will traverse the layer in a shorter period of time. Such increased mobility enables use of an element, for example, in a high speed copier employing high-intensity, short-duration imagewise exposure (commonly also referred to as flash exposure), wherein the time it will take for the element to properly discharge, and, thus, the length of the period needed between the end of the exposure step and the beginning of the toner image development step, is determined by the level of electron-mobility within the element. The higher the mobility is, the shorter is the necessary waiting period between exposure and development, and the greater is the number of copies that can be made in a given amount of time.

Thus, there is a continuing need for electrophotographic elements containing new chemical compounds that will serve well as electron-transport agents in the elements without imparting undesirably high dark decay characteristics thereto and that will also exhibit improved solubility or dispersibility in coating solvents of choice and improved compatibility with polymeric film-forming binders of choice.

The present invention meets this need.

The present invention provides electrophotographic elements containing electron-transport agents comprising new chemical compounds, which are derivatives of 4H-thiopyran-1,1-dioxides and have the structure



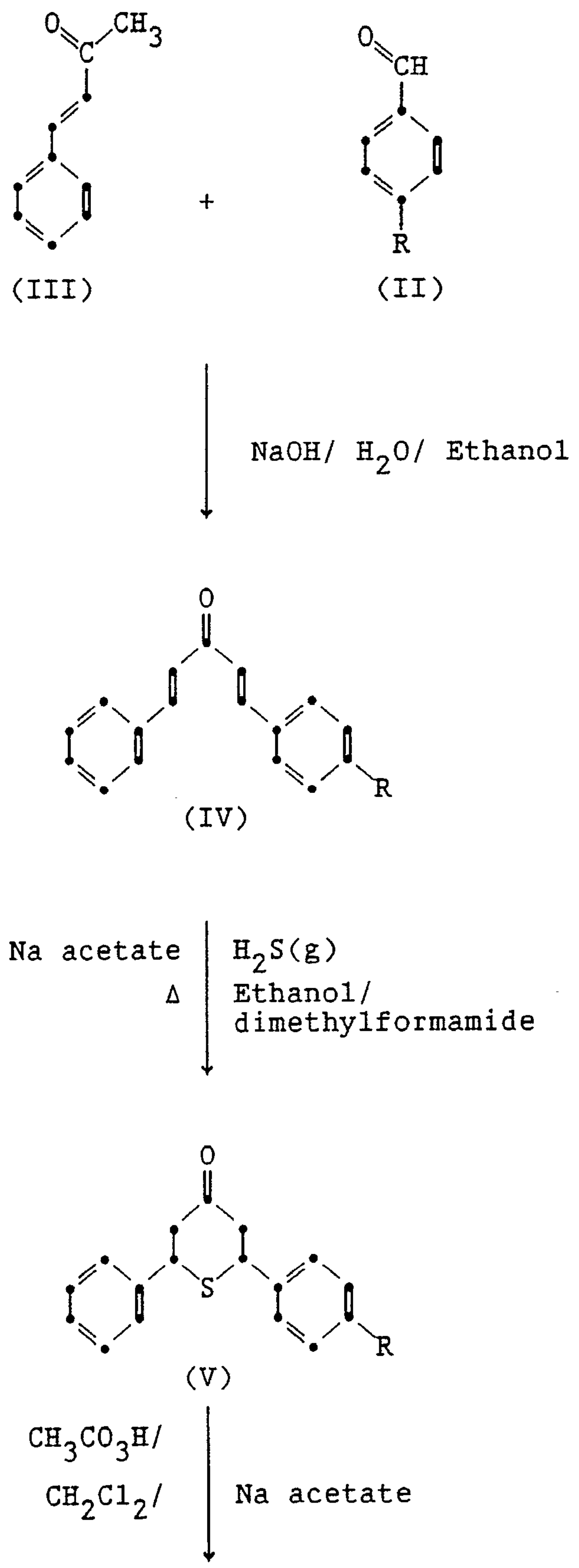
(I)

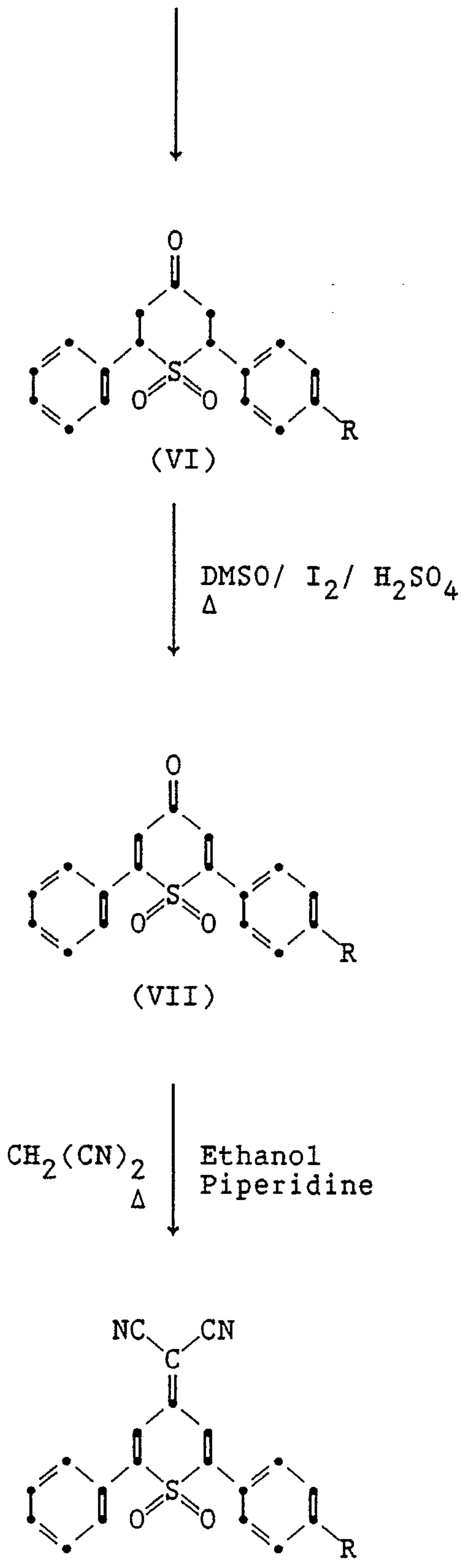
wherein R is alkyl having 1 to 25 carbon atoms.

The new chemical compounds useful as electron-transport agents in elements of the invention have better solubility or dispersibility in many coating solvents and in many film-forming polymeric binders, that are useful to form one or more layers in the inventive electrophotographic elements, than do many previously known electron-transport agents (e.g., those specifically revealed in U.S. Patent 4,514,481). In the inventive electrophotographic elements, the chemical compounds serve as electron-transport agents with good capability of accepting and transporting electrons generated by radiation-activated charge-generation materials in the elements, and they do not impart unacceptably high dark decay properties to the elements. In fact, because of their better compatibility with polymeric binders, the new chemical compounds can be included in electrophotographic elements at concentrations high enough to yield inventive elements that exhibit better electron-transport mobility and even better electrophotographic sensitivity (i.e., less actinic radiant energy is needed to properly discharge the elements) than elements that employ electron-transport agents such as those revealed in U.S. Patent 4,514,481.

The chemical compounds of Structure (I) utilized as electron-transport agents in electrophotographic elements of the invention can be prepared from readily available starting materials, for example, by condensation of the appropriate p-R-substituted benzaldehyde (II) with benzalacetone (III) to yield 1-phenyl-5-(4-R-phenyl)-1,4-pentadiene-3-one (IV); (IV) can then be reacted with hydrogen sulfide to yield 2-phenyl-6-(4-R-phenyl)-2,3,5,6-tetrahydro-4H-thiopyran-4-one (V), which can be reacted with peracetic acid to form the sulfone (VI); (VI) can then be oxidized with dimethylsulfoxide to yield 2-phenyl-6-(4-R-phenyl)-4H-thiopyran-4-one-1,1-dioxide (VII); (VII) is then converted to 4-dicyanomethylene-2-phenyl-6-(4-R-phenyl)-4H-thiopyran-1,1-dioxide (I) by Knoevenagel condensation with malononitrile. This synthetic scheme can be illustrated as follows.

Scheme I


O
CH3
C
+
O
CH
R
(III)
(II)
NaOH/ H2O/ Ethanol
O
R
(IV)
Na acetate
Δ
H2S(g)
Ethanol/
dimethylformamide
O
S
R
(V)
CH3CO3H/
CH2Cl2/
Na acetate



(VI)

(VII)

(I)

Further details of this synthetic scheme are presented in the specific Preparations, infra.

Although R can be C_1-C_{25} alkyl, in preferred embodiments R is C_1-C_{10} alkyl, because such Structure

(I) compounds have a higher electron-accepting functionality-to-mass ratio; i.e., they are more efficient electron-transporters per unit mass. In this regard, the most preferred embodiment contains the Structure (I) compound wherein R is methyl.

To illustrate the solubility/compatibility advantages of Structure (I) compounds, for example, in regard to solubility at room temperature (ca. 22° C) in dichloromethane (a solvent preferred for use in solvent-coating polymeric layers of electrophotographic elements), the preferred Structure (I) compound, 4-dicyanomethylene-2-phenyl-6-(4-tolyl)-4H-thiopyran-1,1-dioxide, has unexpectedly been found to be soluble up to a concentration of at least 6% by weight, and, in regard to compatibility at room temperature with poly[4,4'-(2-norbornylidene)diphenylene terephthalate-co-azelate] (a preferred film-forming polyester for use in preparing polymeric layers of electrophotographic elements), the same preferred Structure (I) compound has unexpectedly been found to be compatible therewith up to a concentration of at least 55% by weight. In comparison, the limit of solubility of 4-dicyanomethylene-2,6-diphenyl-4H-thiopyran-1,1-dioxide (Compound 3 of Table I of U.S. Patent 4,514,481, a compound outside the scope of present Structure (I)) in dichloromethane is about 4% by weight, and its limit of compatibility with the polyester identified above is about 35% by weight. Thus, the Structure (I) compound is seen to have significant solubility/ compatibility advantages over the prior art compound.

These unexpected advantages are also apparent in comparison to other similar, but previously unknown, compounds just outside the scope of Structure (I). For example the solubility and compatibility limits, in regard to the same solvent and polyester as above, are less than 3 weight % and less than 30 weight %, respectively, for the compound, 4-dicyanomethylene-2-(4-chlorophenyl)-6-(4-tolyl)-4H-thiopyran-1,1-dioxide, and are about 3.3 weight % and about 20 weight %, respectively, for 4-dicyanomethylene-2-phenyl-6-(4-methoxyphenyl)-4H-thiopyran-1,1-dioxide (both compounds being outside the scope of Structure (I)).

The new electrophotographic elements of the invention can be of various types, all of which contain one or more of the chemical compounds of Structure (I) described above to serve as electron-transport agents in the elements. The various types of inventive elements include both those commonly referred to as single layer or single-active-layer elements and those commonly referred to as multiactive, multilayer, or multi-active-layer elements.

Single-active-layer elements are so named, because they contain only one layer that is active both to generate and to transport charges in response to exposure to actinic radiation. Such elements typically comprise at least an electrically conductive layer in electrical contact with a photoconductive layer. In single-active-layer elements of the invention, the photoconductive layer contains a charge-generation material to generate electron/hole pairs in response to actinic radiation and an electron-transport material, comprising one or more of the chemical compounds of Structure (I) described above, which is capable of accepting electrons generated by the charge-generation material and transporting them through the layer to effect discharge of the initially uniform electrostatic potential. The photoconductive layer is electrically insulative, except when exposed to actinic radiation, and sometimes contains an electrically insulative polymeric film-forming binder, which may itself be the charge-generating material or may be an additional material which is not charge-generating. In either case the electron-transport agent is dissolved or dispersed as uniformly as possible in the binder film.

Multiactive elements are so named, because they contain at least two active layers, at least one of which is capable of generating charge (i.e., electron/hole pairs) in response to exposure to actinic radiation and is referred to as a charge-generation layer (hereinafter also referred to as a CGL), and at least one of which is capable of accepting and transporting charges generated by the charge-generation layer and is referred to as a charge-transport layer (hereinafter also referred to as a CTL). Such elements typically comprise at least an electrically conductive layer, a CGL, and a CTL. Either the CGL or the CTL is in electrical contact with both the electrically conductive layer and the remaining CGL or CTL. The CGL contains at least a charge-generation material; the CTL contains at least a charge-transport agent; and either or both layers can contain an electrically insulative film-forming polymeric binder. In multiactive elements of the invention the charge-transport agent is an electron-transport agent comprising one of the chemical compounds of Structure (I) described above.

Single-active-layer and multiactive electrophotographic elements and their preparation and use, in general, are well known and are described in more detail, for example, in U.S. Patents 4,701,396; 4,666,802; 4,578,334; 4,719,163; 4,175,960; 4,514,481; and 3,615,414. The only essential difference of electrophotographic elements of the present invention from generally known elements is that the new elements contain chemical compounds of Structure (I) as electron-transport agents.

In preparing single-active-layer electrophotographic elements of the invention, the components of the photoconductive layer, including any desired addenda, can be dissolved or dispersed together in a liquid and can be coated on an electrically conductive layer or support. The liquid is then allowed or caused to evaporate from the mixture to form the permanent layer containing from 10 to 70 percent (by weight) of the electron-transport agent and from 0.01 to 50 weight percent of the charge-generating material. Included among many useful liquids for this purpose are, for example, aromatic hydrocarbons such as benzene, toluene, xylene and mesitylene; ketones such as acetone and butanone; halogenated hydrocarbons such as dichloromethane, trichloroethane, chloroform, and ethylene chloride; ethers, including ethyl ether and cyclic ethers such as tetrahydrofuran; other solvents such as acetonitrile and dimethylsulfoxide; and mixtures thereof.

In preparing multiactive electrophotographic elements of the invention, the components of the CTL can be similarly dissolved or dispersed in such a liquid coating vehicle and can be coated on either an electrically conductive layer or support or on a CGL previously similarly coated or otherwise formed on the conductive layer or support. In the former case a CGL is thereafter coated or otherwise formed (e.g., by vacuum-deposition) on the CTL. The CTL will usually contain from 10 to 70 weight percent of the electron-transport agent, although concentrations outside that range may be found to be useful in some cases.

Various electrically conductive layers or supports can be employed in electrophotographic elements of the invention, such as, for example, paper (at a relative humidity above 20 percent); aluminum-paper laminates; metal foils such as aluminum foil, zinc foil, etc.; metal plates such as aluminum, copper, zinc, brass and galvanized plates; vapor deposited metal layers such as silver, chromium, vanadium, gold, nickel, aluminum and the like; and semiconductive layers such as cuprous iodide and indium tin oxide. The metal or semiconductive layers can be coated on paper or conventional photographic film bases such as poly(ethylene terephthalate), cellulose acetate, polystyrene, etc. Such conducting materials as chromium, nickel, etc. can be vacuum-deposited on transparent film supports in sufficiently thin layers to allow electrophotographic elements prepared therewith to be exposed from either side.

Any charge-generation material can be utilized in elements of the invention. Such materials include inorganic and organic (including monomeric organic, metallo-organic and polymeric organic) materials, for example, zinc oxide, lead oxide, selenium, phthalocyanine, perylene, arylamine, polyarylalkane, and polycarbazole materials, among many others.

When solvent-coating a photoconductive layer of a single-active-layer element or a CGL and/or CTL of a multiactive element of the invention, a film-forming polymeric binder can be employed. The binder may, if it is electrically insulating, help to provide the element with electrically insulating characteristics. It also is useful in coating the layer, in adhering the layer to an adjacent layer, and when it is a top layer, in providing a smooth, easy to clean, wear-resistant surface.

The optimum ratio of charge-generation or charge-transport material to binder may vary widely depending on the particular materials employed. In general, useful results are obtained when the amount of active charge-generation and/or charge-transport material contained within the layer is within the range of from 0.01 to 90 weight percent, based on the dry weight of the layer.

Representative materials which can be employed as binders in charge-generation and charge-transport layers are film-forming polymers having a fairly high dielectric strength and good electrically insulating properties. Such binders include, for example, styrene-butadiene copolymers; vinyl toluene-styrene copolymers; styrene-alkyd resins; silicone-alkyd resins; soya-alkyd resins; vinylidene chloride-vinyl chloride copolymers; poly(vinylidene chloride); vinylidene chloride-acrylonitrile copolymers; vinyl acetate-vinyl chloride copolymers; poly(vinyl acetals), such as poly(vinyl butyral); nitrated polystyrene; poly(methylstyrene); isobutylene polymers; polyesters, such as poly[ethylene-co-alkylenebis(alkyleneoxyaryl)-phenylenedicarboxylate]; phenolformaldehyde resins; ketone resins; polyamides; polycarbonates; polythiocarbonates; poly[ethylene-co-isopropylidene-2,2-bis(ethyleneoxyphenylene)terephthalate]; copolymers of vinyl haloacrylates and vinyl acetate such as poly(vinyl m-bromobenzoate-co-vinyl acetate); chlorinated poly(olefins), such as chlorinated poly(ethylene); and polyimides, such as poly[1,1,3-trimethyl-3-(4'-phenyl)-5-indane pyromellitimide].

Binder polymers should provide little or no interference with the generation or transport of charges in the layer. Examples of binder polymers which are especially useful include bisphenol A polycarbonates and polyesters such as poly[4,4'-norbornylidene)diphenylene terephthalate-co-azelate].

CGL's and CTL's in elements of the invention can also optionally contain other addenda such as leveling agents, surfactants, plasticizers, sensitizers, contrast-control agents, and release agents, as is well known in the art.

Also, elements of the invention can contain any of the optional additional layers known to be useful in electrophotographic elements in general, such as, e.g., subbing layers, overcoat layers, barrier layers, and screening layers.

The following preparations and examples are presented to further illustrate some specific electrophotographic elements of the invention and chemical compounds useful as electron-transport agents therein.

Preparation A: 4-Dicyanomethylene-2-phenyl-6-(4-tolyl)-4H-thiopyran-1,1-dioxide (Compound I-A)

Benzalacetone (Compound III of Scheme I, supra) was purchased from Aldrich Chemical Co., U.S.A.

A mixture of 146.0 g (1.0 mole) of benzalacetone and 122.0 g (1.02 mole) 4-tolualdehyde (Compound II-A) was dissolved in 750 ml ethanol, and the solution was diluted with 200 ml water. With stirring, at room temperature, all at once, 100 ml of 10% NaOH solution were added. After 1.5 hours the thick, cream precipitate was filtered off, resuspended in 1 liter 20% ethanol in water, refiltered, sucked dry and recrystallized from ethanol/ethyl acetate to give 180 g of 1-phenyl-5-(4-tolyl)-1,4-pentadiene-3-one (Compound IV-A). The product structure was confirmed by field desorption mass spectrometry (FDMS) and infrared (IR) spectrometry.

In a 2 liter round-bottom flask fitted with an electrically driven stirrer, a gas inlet tube, and a reflux condenser connected to an anti-suckback trap, and to an H_2S trap, was placed a mixture of 180 g (0.73 mole) of Compound IV-A, 51.4 g (0.63 mole) anhydrous sodium acetate, 140 ml dimethylformamide (DMF) and 370 ml ethanol. The mixture was stirred and heated on a steam bath while passing in a brisk stream of H_2S gas. When thin-layer chromatography (TLC) showed complete conversion of colored starting material to a colorless product, the reaction mixture was poured into an excess of cold water. The product was extracted into dichloromethane (DCM), dried (over $MgSO_4$), filtered, and evaporated down. The pasty product, dried in air weighed 213.5 g. It was confirmed to be 2-phenyl-6-(4-tolyl)-2,3,5,6-tetrahydro-4H-thiopyran-4-one (Compound V-A) by FDMS.

A mixture of 213.5 g crude Compound V-A, and 16.5 g of anhydrous sodium acetate in 825 ml DCM was cooled in ice, and treated dropwise with 200 ml commercial peracetic acid. When the ice bath was lowered, and the addition of peracid was more brisk, the DCM refluxed gently. In the same way another 225 ml of peracid were added and the mixture was let stand overnight. The next day another 50 ml peracid were added and the mixture was refluxed for 0.5 hr. The reaction mixture was poured into an excess of water and neutralized, first with 20% NaOH solution and then with solid sodium bicarbonate. The organic precipitate was extracted into dichloromethane, dried (over $MgSO_4$), filtered and evaporated down. The yellow residual syrup, comprising 2-phenyl-6-(4-tolyl)-2,3,5,6-tetrahydro-4H-thiopyran-4-one-1,1-dioxide (Compound VI-A), was used directly in the next step.

The crude product (Compound VI-A) from the preceding step was dissolved in 450 ml dimethylsulfoxide (DMSO) and 3.0 g of solid iodine were added. The mixture was warmed slightly and 5 ml concentrated sulfuric acid were added in a thin stream. The flask was lightly stoppered and heated on a steam bath. The mixture was left warm overnight, and then another 1.0 g of iodine was added and the mixture was reheated on a steam bath. TLC then showed complete conversion of starting material to a single yellow product. The reaction mixture was diluted into water, neutralized with solid sodium bicarbonate, and extracted with DCM. This solution, on being dried (over $MgSO_4$), filtered, and evaporated down, readily yielded a crystalline solid which was recrystallized from ethanol/ethyl acetate to give 156 g of yellow solid confirmed by spectrometric analysis to be 2-phenyl-6-(4-tolyl)-4H-thiopyran-4-one-1,1-dioxide (Compound VII-A).

A mixture of 277 g Compound VII-A, and 140 g redistilled malononitrile in 1.8 liters ethanol was heated gently on a steam bath and 16 ml piperidine were added in drops, with stirring. The mixture immediately turned a deep brick-red. It was heated to gentle reflux and all the solids went into solution, but shortly a precipitate formed and the mixture became thick. TLC showed the formation of a bright-yellow product at high R_f and a number of impurities. The mixture was chilled and the solid was filtered off, resuspended in 400 ml clean ethanol and refiltered. The solid was recrystallized three times from ethanol/toluene and then appeared homogeneous by TLC. The yield of yellow solid product was 148 g.

The product was confirmed to be 4-dicyanomethylene-2-phenyl-6-(4-tolyl)-4H-thiopyran-1,1-dioxide (Compound I-A) by nuclear magnetic resonance (NMR) and IR spectroscopies, high-performance liquid-chromatography (HPLC), and FDMS. Melting point = 186.5 - 187.5° C.

In the following Examples, the structure, preparation, and performance of some electrophotographic elements within the scope of the present invention are illustrated. Performance is illustrated in regard to electrophotographic sensitivity and dark decay properties.

In illustrating electrophotographic sensitivity in the Examples, the element was electrostatically corona-charged to an initial positive potential (usually about 500 volts) and then exposed to actinic radiation (radiation having peak intensity at a wavelength to which the charge-generation material in the element is sensitive in order to generate electron/hole pairs) in an amount sufficient to photoconductively discharge the initial voltage down to levels of 250 V and 100 V. Electrophotographic sensitivity was measured in terms of the amount of incident actinic radiant energy (expressed in ergs/cm^2) needed to discharge the initial voltage down to the desired level. The lower the amount of radiation needed to achieve the desired degree of discharge, the higher is the electrophotographic sensitivity of the element, and vice versa.

In illustrating dark decay properties in the Examples, the rate of dissipation of the initial voltage (expressed in V/s, i.e., volts per second) was measured while the element remained in darkness (i.e., before any exposure to actinic radiation). This was accomplished by measuring the initial voltage and the voltage remaining on the element after 2 seconds in darkness and dividing the difference by 2. The lower the rate of discharge in darkness, the better is the dark decay property of the element, i.e., the better is the element's ability to retain its initial potential before exposure.

In the table of performance data in the Examples, "Electron-transport agent", refers to the chemical compound incorporated in the CTL of an electrophotographic element to serve as an electron-transport agent. "Wt%" refers to the percent by weight of electron-transport agent employed, based on the total weight of polymeric binder and electron-transport agent included in the solution used to coat the CTL of the element. "V_o" refers to the uniform positive potential (in volts) on the element, after it was charged by corona-charging and after any dark decay, such potential having been measured just prior to any exposure of the element to actinic radiation. "DD" refers to the rate of dark decay of the element, prior to exposure to actinic radiation, measured in volts per second (V/s) as described above. "E(250 V)" refers to the amount of incident actinic radiant energy (expressed in ergs/cm^2) that was needed to discharge the element down to a level of 250 volts. "E(100 V)" refers to the amount of incident actinic radiant energy (expressed in ergs/cm^2) that was needed to discharge the element down to a level of 100 V.

Examples 1-3 and Control A

Electrophotographic elements of the invention containing various concentrations of new electron-transport agent in their CTL's were prepared as follows.

A conductive layer-coated support was prepared by vacuum-depositing a thin conductive layer of aluminum onto a 178 micrometer thickness of poly(ethylene terephthalate) film.

A charge-generation layer (CGL) was prepared by dispersing the charge-generation material, titanyl tetrafluorophthalocyanine (described more extensively in U.S. Patent 4,701,396), in a solution of a polymeric binder, comprising a polyester formed from 4,4'-(2-norbornylidene)diphenol and terephthalic acid:azelaic acid (40:60 molar ratio), in dichloromethane (the weight ratio of charge-generation material:binder being 2:1), ball milling the dispersion for 60 hours, diluting with a mixture of dichloromethane (DCM) and 1,1,2-trichloroethane (TCE) (to yield a final DCM:TCE weight ratio of 80:20) to achieve suitable coating viscosity, coating the dispersion onto the conductive layer, and drying off the solvent to yield a CGL of 0.5 micrometer thickness.

A coating solution for forming a charge-transport layer (CTL) was then prepared comprising 10 weight percent solids dissolved in dichloromethane. The solids comprised the electron-transport agent, Compound I-A, prepared as in Preparation A above and a polymeric binder comprising a polyester formed from 4,4'-(2-norbornylidene)diphenol and terephthalic acid:azelaic acid (40:60 molar ratio). The concentration of electron-transport agent was different for each Example, as noted in Table I. The solution was then coated onto the CGL and dried to form the CTL on the CGL. The combined thickness of CGL and CTL was in the range of 7 to 10 micrometers.

The CTL for Control A was prepared and coated in the same manner, except that the electron-transport agent comprised 4-dicyanomethylene-2,6-diphenyl-4H-thiopyran-1,1-dioxide (Compound 3 of Table I of U.S. Patent 4,514,481, a compound outside the scope of present Structure (I))

Each of the resultant electrophotographic elements was then corona-charged to a uniform positive potential.

Dark decay rate of the initial potential was measured for each element.

Each of the uniformly charged elements was subjected to simulated imaging exposure by exposing it through the outer surface of the CTL to radiation having a wavelength of about 820 nanometers (nm) (to which the charge-generation material is sensitive, in order to generate electron/hole pairs in the CGL) at a rate of 2.68 ergs of radiant energy per square centimeter of element surface per second (2.68 ergs/cm^{2}s), and E(250 V) and E(100 V) were measured for each element.

Results are presented in Table I.

Table I

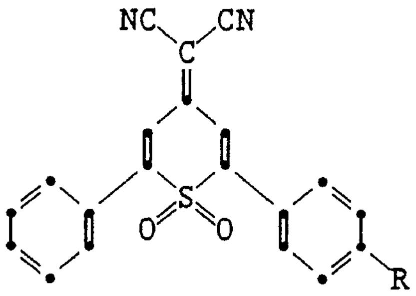
Example	Electron-transport agent	Wt.%	V_o (V)	DD (V/s)	E(250 V) (ergs/cm^2)	E(100 V) (ergs/cm^2)
Control A	4-dicyanomethylene-2,6-diphenyl-4H-thiopyran-1,1-dioxide	30*	464	15	4.4	15.5
1	4-dicyanomethylene-2-phenyl-6-(4-tolyl)-4H-thiopyran-1,1-dioxide	31.2*	478	16	4.0	12.5
2	"	40	454	20	4.3	15.1
3	"	50	456	17	3.9	11.4

* These Wt.%'s represent equimolar concentrations of 0.87 mmole/g.

The data in Table I show that electrophotographic elements of the invention, containing new electron-transport agents, exhibit similar dark decay properties and even better electrophotographic sensitivity, compared to an element containing a known electron-transport agent. The data also shows that a Structure (I) compound can be coated at a relatively high concentration in the coating solution (e.g., 5 weight %) to yield a relatively high, yet still compatible, concentration in the CTL binder polymer (e.g., 50 weight %).

Claims

1. An electrophotographic element containing an electron-transport agent, characterized in that the electron-transport agent comprises a chemical compound having the structure



wherein R is alkyl having 1 to 25 carbon atoms.

2. The electrophotographic element of claim 1, wherein R is methyl.

3. The electrophotographic element of claim 1, wherein the element is a multiactive element comprising: an electrically conductive layer; a charge-generation layer comprising a charge-generating material; and a charge-transport layer comprising a polymeric film containing the electron-transport agent of claim 1.

European
Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 12 4455

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
D,X	US-A-4 514 481 (M. SCOZZAFAVA et al.) * Abstract; column 1, line 51 - column 2, line 49; column 7, line 27 - column 7, line 40; claims 1-3 * — — — — —	1-3	G 03 G 5/06
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			G 03 G

The present search report has been drawn up for all claims

Place of search	Date of completion of search	Examiner
The Hague	22 February 91	HILLEBRECHT D.A.O.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same catagory
A : technological background
O : non-written disclosure
P : intermediate document
T : theory or principle underlying the invention

E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

&: member of the same patent family, corresponding document